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Impact of nano-silica and micro-silica incorporation on strength and chloride resistance of high-performance concrete

ABSTRACT

In this study, the effect of Nano-silica (NS) and Micro-silica (MS) on the mechanical properties and chloride resistance of HPC is investigated. The novelty in this research is the analysis of the synergism between NS and MS pozzolana and finding the optimum ratio of hybrid dosages. In order to conduct the experiment, four concrete mixes were produced including control (M1) and three modified (M2, M3, and M4) by replacing the binder with cement. In M2, M3, and M4 the percentages of MS and NS in the mix are 1+10%, 2+20%, and 3+30%, respectively. In fresh state, the concrete mixes were characterized by slump test, compaction test, and Vee-Bee test. For characterization in the hardened state, compressive, split tensile, and flexural strengths were measured in 7, 14, 28, 56, and 90 days of age. For testing the chloride resistance, $AgNO_3$ and Volhard titration tests were conducted after 28, 90, and 180 days. It was observed that the increase in percentage of silicas led to less workability of fresh concrete but increased strength and chloride resistance of the concrete.The optimal result was obtained from M3, which recorded a 20.6% enhancement in the compressive strength together with better resistance to the penetration of chlorides. These results show that the application of both NS and MS is effective in the improvement of the structural properties of HPC.

Keywords: *Nano-silica, Micro-silica, High-performance concrete, Chloride resistance, Pozzolanic reaction*

1. INTRODUCTION

Nano-silica (NS) and micro-silica (MS) The best dosage is usually about 2%, beyond which mechanical properties could plateau or even decrease due to agglomeration and higher water demand [1]. 2% NS enhanced the 28 days compressive strength by more than 20% and substantially improved **the** pore structure, achieving the highest compactness and strength. NS also enhances early-age strength development because it has a nucleation effect [2, 3].

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MS, commonly utilized at 5–15% replacement levels, continuously enhances compressive strength and tensile strength, with 15% MS achieving as much as 28% greater compressive strength at 28 days than control mixes. [4] Due to its coarser particle size relative to NS, the impact of MS is more significant when applied at higher rates, and it is especially useful in enhancing long-term strength and durability [5]. MS decreases chloride permeability and water absorption as well, although its impact is typically less than that of NS at the same dosage. At higher replacement levels of 10–15%, however, MS has been shown to obtain sizable decreases in chloride penetration and enhance resistance to hostile environments. [6]

NS considerably decreases chloride ion permeability, water intake, and capillary suction, largely through improving the pore structure and enhancing the density of the C-S-H gel [7]. Best NS dosages by 1–3% can decrease chloride penetration by as much as 73% and increase electrical resistivity, suggesting improved strength. NS also advances resistance to freeze-thaw, carbonation, and sulfate attack [8, 9].

The blend of NS and MS provides the lowest chloride permeability and highest electrical resistivity, which shows the best durability [10]. Ternary mixtures show greater denser pore structure, lower overall porosity, and smaller average pore size, all of which together hinder the intrusion of aggressive agents [11, 12]. This synergy is especially useful for structures subjected to marine or deicing conditions. Combining NS and MS in ternary blends creates a synergistic effect, further improving mechanical properties than what is possible with either material individually [5, 13]. A 2.5% NS + 2.5% MS blend had the highest compressive strength and lowest porosity due to enhanced particle packing and denser microstructure. The combination also optimizes the ITZ and lowers the demand for water over the use of NS individually [14].

Addition of NS always enhances compressive strength. Mixes containing 2% NS at 28 days exhibit compressive strengths of 75–82 MPa whereas control HPC exhibits 65–70 MPa. Agglomeration at levels of NS above 5% lowers strength by as much as 5–7%. [9, 15] In the case of MS, 10% by weight of cement replacement always enhances compressive strength from control level of 65 MPa to 72–76 MPa at 28 days. [7, 16] Increased replacement levels (15–20%) decrease workability, with losses of strength by 2–5 MPa reported. [17] Flexural strength of HPC containing 2% NS + 10% MS is 7.0–7.5 MPa, whereas that of control concrete is 6.0–6.2 MPa. [19] NS and MS enhance splitting tensile strength as well. For example, mixes containing 2% NS and 10% MS obtain 5.2–5.5 MPa, while control mixes obtain 4.4–4.6 MPa. [20, 21] Ternary mixtures of NS + MS with other pozzolans have compressive strengths of 85–90 MPa at 28 days, against 70 MPa in control, which is an improvement of up to 25–30%. [22, 23].

Best range is NS 1–3% of binder weight, with 2% NS producing compressive strength of around 80 MPa and RCPT below 1000 Coulombs. [24] Optimum performance at 10% MS replacement, producing compressive strength of 75 MPa and chloride permeability around 1200 Coulombs. [25] A blend of 2% NS and 10% MS always provides the optimum balance, with compressive strengths of 80–82 MPa, splitting tensile strength of 5.3 MPa, flexural strength of 7.3 MPa, and chloride permeability values of 700–800 Coulombs. [26] MS, as an industrial by-product, saves cement consumption by 10–15%, which in turn saves 80–100 kg CO_2/ton of concrete. Even though NS is more expensive roughly 5–8 times the cost of cement per kg, it needs only small amounts (1–3%). [27, 28] Both NS and MS need higher water content. Without superplasticizers, the slump can fall from 180 mm (control) to 120–130 mm. High-range water-reducing admixtures provide workability. [29]

The literature agrees on maximum concentrations of 1–3% for NS and 5–15% for MS. Concentrations above these may result in particle agglomeration, excessive water requirement, and reduced mechanical performance. Mechanical dispersion methods and the incorporation of superplasticizers are vital to achieve full potential for NS and MS. Cement replacement with NS and MS not only improves performance but also reduces carbon emissions from concrete, leading to greener construction methods. Utilization of industrial by products and nanomaterials is consistent with green building goals. Though NS and MS benefits have long been reported, there are issues of ensuring a uniform dispersion, determining long term durability under field conditions, and maximizing cost effectiveness. The purpose of this research is to identify the effects of the combination of NS and MS with chloride attacks, their performance in high performance concrete, and their performance under harsh environmental exposures.The present study is novel in its systematic assessment of the combined multiscale effect of nano-silica and micro-silica in HPC, with particular emphasis on identifying the optimum hybrid replacement level for maximizing both mechanical strength and chloride resistance over long-term curing durations.

2. MATERIALS

Physical and chemical properties of cement and pozzolonic admixture significantly contribute to the performance of concrete. Properties of cement used in this research were as follows; density ratio of 3.15, consistency of 30.12%, initial setting time of 39 min, setting time of 5hr3 min, adequate workability and curing period. Expansion ratio due to soundness property was 1.01mm, which is suitable for dimensional stability. Compressive strength of 43.42MPa after 28 days makes the suitability of its usage for structural applications. Chemical analysis shows 61.63% CaO, 21.03% SiO_2, 6.04% Al_2O_3, 5.98% Fe_2O_3 and 1.81% SO_3 along with traces of MgO (1.43%), alkali (0.81%) and LOI (1.03%) all within permissible limits.

Fine aggregate was found to have relative density as 2.61, fineness modulus as 2.66, bulk density as 1669 kg/m3, moisture content as 0.11% and water absorption as 0.74%. This gives an indication of its ability to display good workability without compromising on its strength. The coarse aggregate showed a relative density of 2.79, fineness modulus of 6.16, void percentage of 36.89%, crushing value of 23.74% and water absorption of 0.5%. Its angular shape allowed for better inter particle bonding and low crushing value denotes high strength.

Micro silica, relative density of 2.65, and fineness modulus of 2.1 provided ultrafine particles that filled pores, minimized porosity, and enhanced strength. Its minimum water absorption (0.24%), bulk density of 243 kg/m³, and very small moisture content (0.05%) provided it as the best for high performance use. Nano silica, with 2.2 g/cm³ density, 1.1 specific gravity, 250 nm particle size, and 160 m²/g surface area, showed outstanding reactivity because it is amorphous in nature, which increases hydration and produces more C–S–H gel.

Application of superplasticizer (Forsoc ComplastSP 430), a brown liquid having specific gravity of 1.18, enhanced workability further by decreasing water requirement and providing dense, flowable mix. Together, the physical as well as chemical characteristics of cement, aggregates, and additives provide for development of long lasting, high strength, and sustainable high performance concrete.

The aggressive chloride environment was selected to simulate severe exposure conditions commonly encountered in marine structures, coastal regions, bridge decks, and reinforced concrete exposed to de-icing salts. Chloride ingress is one of the primary causes of reinforcement corrosion and premature deterioration in concrete structures, making it a critical parameter for evaluating the long-term durability of high-performance concrete. To assess chloride resistance, two complementary methods were employed.

The silver nitrate ($AgNO_3$) visual inspection method was used to identify the depth of chloride penetration through colorimetric indication, providing a rapid qualitative assessment of chloride ingress. In addition, the Volhard titration method was adopted to quantitatively determine chloride concentration within the concrete specimens. The combined use of these methods enables both visual and analytical evaluation of chloride penetration behavior, thereby providing a comprehensive understanding of the resistance offered by nano-silica and micro-silica modified concrete mixes under aggressive chloride exposure.

3. EXPERIMENTAL INVESTIGATION

3.1 Fresh concrete properties

Fresh concrete workability was determined by the standard slump cone test. The cone was loaded in three layers, tamping each layer 25 times with a standard tamping rod. The cone was then lifted vertically and the vertical settlement of the concrete (slump) measured in millimeters. Replicates for each mix were repeated three times and the average value reported.

The compaction factor was determined with the two cylinder apparatus. The lower cylinder was charged with concrete, the top cylinder placed on it and the splitting arrangement utilized to provide a means for concrete to drop into the lower cylinder. The weights of partially and fully compacted concrete were measured and the compaction factor determined as the ratio of the weight after standard compaction to the weight after full compaction. Three replicates were carried out per mix.

In cases of mixes where the slump reported poor flow, the Vee-Bee consistometer was employed to measure the vibration conversion of the paste. A sample was taken to the specified level of slump in the Vee-Bee cup, the vibrating table was initiated and the time for the concrete to transform to a cylindrical shape measured as the Vee-Bee time. Twice for every mix, tests were conducted and the average reported.

3.2. Mechanical properties

Compressive strength was also measured on 150 mm cubes of concrete (or according to relevant national standard) at specified ages (7, 14, 28 and 90 days).Specimens were water cured up to the test age. Loading under displacement control at a constant rate up to failure and maximum load was measured. Strength was calculated by dividing the failure load by loaded area. Three specimens of each age and blend were tested at least, and the mean and standard deviation were given. Cylindrical test specimens (150 mm diameter × 300 mm high) were employed. Specimen was loaded horizontally between platens with timber bearing strips, and load was applied to splitting failure. Tensile strength was determined from the maximum load by the standard splitting formula. Three test specimens for each mix and age were tested, and a mean value was reported.

Flexural strength was determined on prismatic beams (100 × 100 × 700 mm or nominal size) under third-point loading. Beams were tested following standard curing at the ages specified. The applied load at failure was used to calculate the modulus of rupture. At least three beams per mix and age were tested and the mean value employed for comparison.

3.3. Chloride resistance specimen preparation and exposure

For rapid chloride testing, 100 mm cubes of concrete were prepared and cured in water for 28 days. Following the initial curing, specimens for chloride exposure were preconditioned as specified below.

3.3.1. Preconditioning

- ❖ Saturation in lime water: Before exposure, cubes were soaked in saturated lime water until constant mass or until the specimen attained near-saturated surface conditions. Excessive and uncontrolled surface absorption of chloride solution is avoided in this manner that otherwise might be causing bias in penetration measurements.
- ❖ Side sealing: All the faces of each cube were painted with an impermeable epoxy paint with one face left uncovered which was to be exposed to the chloride solution. This provides for unidirectional chloride penetration through the exposed face alone.

3.3.2. Preparation of chloride solution

2.82 M solution of sodium chloride was made by dissolving 165 g of NaCl in 1 litre of distilled water (2.82 mol·L^{-1} × 58.44 g·mol^{-1} = 165 g). The mixture was stirred until everything dissolved and kept in a sealed vessel. Periodically, the concentration of the solution was monitored and replenished or replaced as necessary to allow for stable exposure.The chloride solution was replaced at regular intervals whenever noticeable concentration reduction was observed due to chloride ion absorption by the concrete specimens, ensuring constant chloride concentration and uniform aggressive exposure throughout the test period.

3.3.3. Exposure protocol

Preconditioned and coated cubes were partially submerged with the exposed face towards the solution in the 2.82 M NaCl solution for specified exposure times. Minimum exposure for the purpose of comparison with control concrete was 28 days; extended time spans of 180 days were applied for aging studies. Solution level and temperature were controlled and regulated throughout the exposure period.

3.4. Chloride measurement and sampling methods

- ❖ Visual mapping (silver nitrate spray): One specimen at some chosen exposure intervals was divided into two halves perpendicular to the exposed surface. A 0.1 N silver nitrate ($AgNO_3$) solution was sprayed immediately over the freshly cut surface. Free chloride reacts with silver nitrate to give white silver chloride (AgCl), which indicates the visible front of free chloride penetration. In HVFA concretes or other binders, a brownish tint can suggest the presence of combined chlorides. The penetration depth was taken 1 cm apart over the face, and the mean penetration depth calculated.
- ❖ Drill sampling and quantitative determination (Volhard titration): For quantitative profiling of chloride, powder samples were recovered by drilling perpendicular to the exposed surface at successive depth increments for instance, 0–2 cm, 2–4 cm, 4–6 cm, etc.,. Drill dust from each depth increment was separately collected in clean containers and weighed. Samples were prepared and dissolved in accordance with the Nordtest NT BUILD 208 procedure to extract the chloride into solution for titration.
- ❖ Volhard titration process: Each of the extracted sample solutions' chloride content was determined using the Volhard titration technique. During this back-titration, a slight excess of standard silver nitrate solution is added to precipitate the chloride as AgCl. The rest of the silver is then titrated against standard ammonium thiocyanate to the endpoint, which is a shift from milky white to a pale red color after the ferric indicator or according to lab procedure has been added. The reagent volumes and normalities used are accurately noted.

4. RESULTS AND DISCUSSION

4.1. Fresh Concrete Properties

Workability of concrete is a key determinant of its ease of placing, compacting, and finishing. This research assesses the workability properties of four mixes of concrete: a control mix (M1) and three modified mixes with different ratios of nano silica and micro silica as partial cement replacements (M2, M3, and M4). The workability was determined based on three conventional tests: slump cone test, compaction factor test, and Vee Bee Consistometer test. The slump values recorded for mixes M1 to M4 showed a decreasing trend. The highest slump of 103 mm was recorded for the conventional concrete (M1), reflecting comparatively high workability. In comparison, modified mixes M2, M3, and M4 recorded slump values of 100 mm, 95 mm, and 91 mm, respectively. The reduction in slump with increasing nano-silica and micro-silica content indicates a decrease in workability.

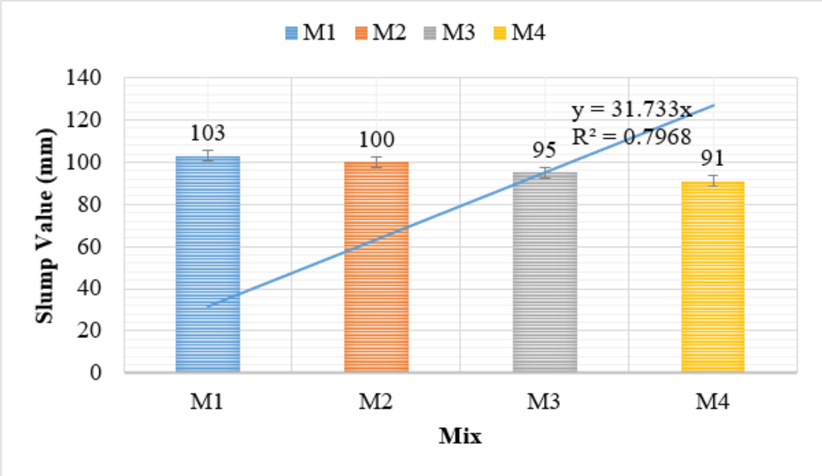


Figure 1. Slump cone

This can be mainly attributed to the fact that nano-silica and micro-silica exhibit a large surface area and pozzolanic activity, thus causing an increase in water requirement due to finer particles and surface absorption. Moreover, since nano-silica and micro-silica have lower density than cement, the larger volume of replacement leads to a higher surface area of the particles, thus decreasing workability. An increase in the percentage of replacement causes a decrease in workability. This trend is shown in Figure 1 below.

The same trend can be seen from the compaction factor values. The compaction factor reduced from 0.858 for M1 to 0.833 for M2, 0.792 for M3, and 0.758 for M4. This steady reduction indicates a decrease in the ease of compaction with an increase in the percentage of cement replacement. Finer pozzolanic material makes the mix more prone to internal friction and cohesion, thus making it less capable of flowing under compaction. The increased replacement volume caused by the lower density of silica-based materials contributes to greater internal particle interaction and friction, thereby reducing flowability under compaction. The compaction factor is a more sensitive measure of low workability, and the results in this case are generally consistent with the observed slump values. Figure 2 illustrates the compaction factor.

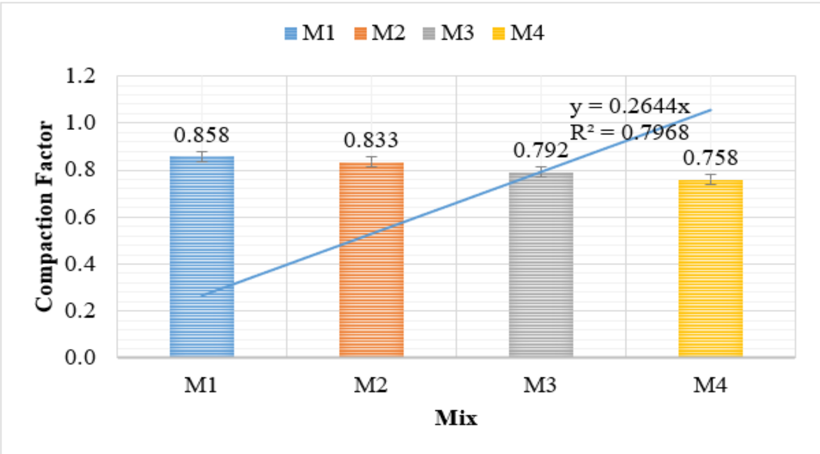


Figure 2. Compaction factor

The Vee Bee Consistometer test results also correlate with the results of the other tests. The time elapsed for complete remolding of the concrete decreased steadily from 2.49 seconds (M1) to 2.42 seconds (M2), 2.30 seconds (M3), and 2.20 seconds (M4). While the drop is not as significant, it is still a measure of reduced workability. A decreased Vee Bee time generally implies increased workability; however, here too the numbers are close enough to suggest that all the mixes have acceptable workability in spite of the drops. This drop might even imply denser mix development with better particle distribution due to the inclusion of nano and micro silica. Figure 3 indicates the results from the Vee Bee consistometer.

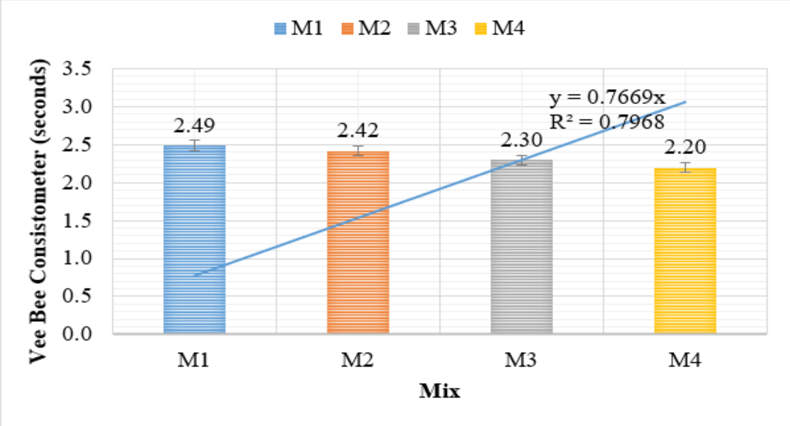


Figure 3. Vee Bee Consistometer

Overall, the results of all three tests clearly show that increasing the proportion of nano-silica and micro-silica as cement replacement causes a measurable reduction in concrete workability. This is expected due to their finer particle size, high surface area, and the volumetric replacement effect arising from their lower densities compared to cement. However, the reduced workability remained within acceptable limits, particularly for M2 and M3. Although M4 exhibited the lowest workability, it may still be suitable for applications where durability is prioritized over ease of placement. Mechanical behavior of concrete has a notable impact on its long-term structural performance.

4.1. Mechanical Properties

In the present study, the impact of nano silica and micro silica as partial cement replacement on the performance characteristics of concrete, namely compressive strength, split tensile strength, and flexural strength, has been analyzed at curing ages of 7, 14, 28, 56, and 90 days. Four different mixes were considered for analysis: Mix ratio M1, control; Mix ratio M2, 1% nano silica and 10% micro silica; Mix ratio M3, 2% nano silica and 20% micro silica; Mix ratio M4, 3% nano silica and 30% micro silica.

The control mix M1 had a strength of 49.92 MPa at 90 days. On the other hand, M2, M3, and M4 had greater 90-day strengths of 57.77 MPa, 60.21 MPa, and 58.59 MPa respectively. The greatest strength was for M3 in all ages of curing, reaching its maximum at 60.21 MPa at 90 days — a 20.6% enhancement from M1. This enhancement is due to the pozzolanic reaction of nano and micro silica, which reinforces the microstructure by producing more calcium silicate hydrate (C-S-H) gel and decreasing porosity. The small particles of nano silica pack in voids efficiently, promoting denser packing. Past the optimum dosage in M4, though, there is a mild strength gain loss compared to M3. This might be because of too much replacement decreasing the cement content too far, minimizing total reactivity by a small amount. The compressive strength is given in Figure 4. The same trend is noted with the split tensile strength values.

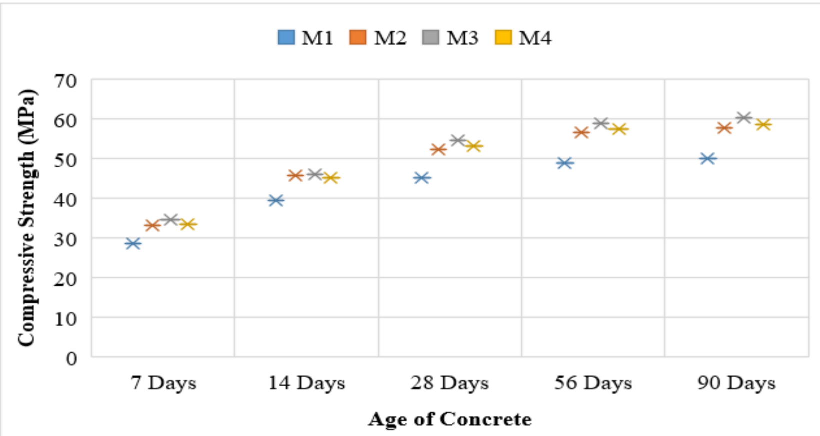


Figure 4. Compressive Strength

M1 reached 4.14 MPa at 90 days, whereas M2, M3, and M4 recorded 4.79 MPa, 4.99 MPa, and 4.86 MPa, respectively. For the fourth time, M3 recorded the maximum values at all ages. The rise in tensile strength is a result of the enhanced bond between aggregates and cementitious matrix with the finer particles and pozzolanic effect of silica additives. Enhanced microstructural integrity and decreased microcracking also lead to enhanced tensile resistance. The split tensile strength is indicated in Figure 5. The evidence of the advantages of nano and micro silica is further provided by the flexural strength data.

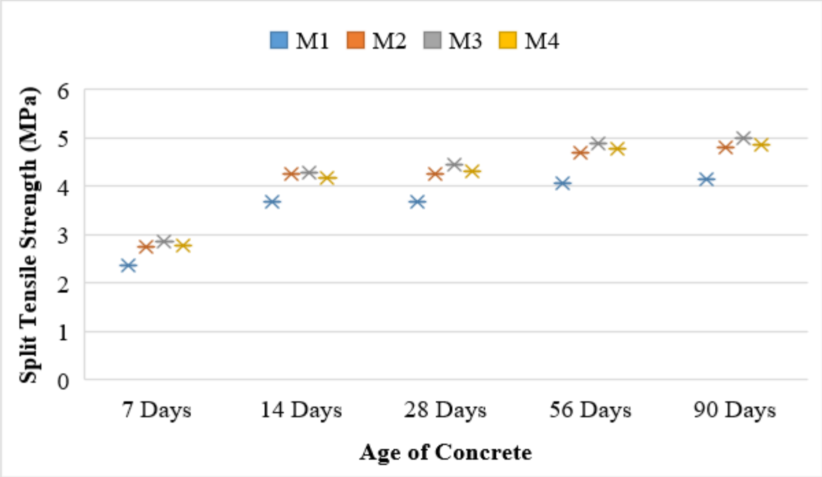


Figure 5. Split tensile strength

M1 exhibited a flexural strength of 6.14 MPa after 90 days, whereas M2, M3, and M4 showed 7.10 MPa, 7.40 MPa, and 7.20 MPa, respectively. The improvement in strength is also similar, with M3 showing the best flexural performance. The improvement is attributed to improved load bearing and resistance to cracks due to the improved pore structure and stronger interfacial transition zone (ITZ). As for compressive strength, the marginal decrease in M4 compared to M3 implies a threshold beyond which more silica has decreasing returns. Figure 6 flexural strength.

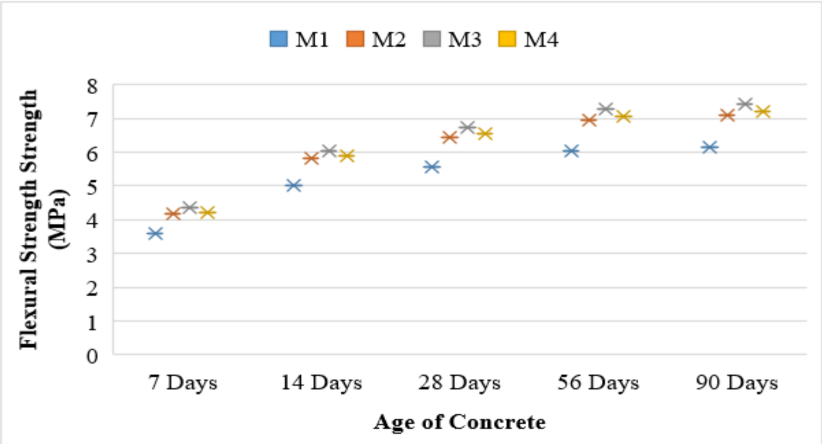


Figure 6. Flexural Strength

All modified blends performed better than the standard mix in all mechanical properties and curing ages. M3 (2% nano + 20% micro silica) performed consistently well, proving it to be the best mix among the ones tested. The combination of nano and micro silica improves the hydration process, densifies the matrix, and enhances strength factors. Yet, a very high replacement (M4) could slightly sacrifice efficiency, perhaps due to lesser cement content or a higher demand for water influencing early hydration.

4.3. Chloride

4.3.1. Free and Combined Chlorides

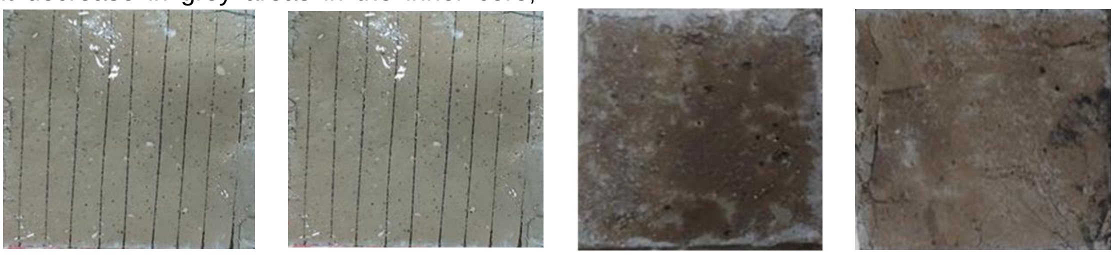
The distribution of free and bound chlorides, as found out by visual inspection through spraying a

0.1N$AgNO_3$ solution on M1, M2, M3, and M4 blends after 28, 90, and 180 days, is represented in Figures 7, 8, and 9, respectively. As evident from Figure 7, free chlorides were mainly found adjacent to the external surface, while bound chlorides were found in the direction of the inner part of the split sample. The mean depth penetrations for blends M2, M3, and M4 were 0.3 cm, 0.54 cm, and 0.8 cm, respectively. The brown color shows the bound chlorides.

After being exposed for 90 days, a general reduction in free chloride concentration was noted in all mixes but not in Mix 1, which demonstrated an average depth of penetration of free chloride to 0.62 cm. In mixes containing nanosilica and microsilica, it was difficult to define the depth of free chlorides owing to reduced workability, increased chloride fixation, and lack of silver chloride precipitation in the surface, as indicated in Figure 8.

With increased time in the chloride environment, all the mixes had enhanced chloride-binding ability. Visual examination after 180 days showed no free chlorides. As indicated by Figure 9, the samples had a darker brown color and an evident decrease in grey areas in the inner core, which demonstrates increased chloride binding compared to previous exposure times.

Chlorides in concrete are classified into free chlorides and combined chlorides. Addition of micro and nano silica content enhances the chloride binding capacity, which decreases the susceptibility of the concrete to corrosion. From the current study, it is clear from the test of visual inspection that chloride binding capacity improves with the length of exposure to a corrosive environment. With greater exposure to a chloride environment, white precipitate (silver chloride) decreases, and combined chloride content increases (brown color). Combined chloride content shows more for higher volume fraction micro silica than for test mixes with smaller volume fractions. The penetration depth of free chloride in test mix decreases with longer exposure time, implying that micro and nano silica pozzolanicity assists in the refinement of pores and, therefore, a denser matrix. The depth (or) of penetration of free chlorides is decreased and is not detected after 90 days of exposure to a chloride environment. This is due to less porosity and more particle packing because of incorporating micro and nano silica.



(a) (b) (c) (d)

Figure 7. Visual inspection at 28 days for (a) M1, (b) M2, (c) M3, and (d) M4

(a) (b) (c) (d)

Figure 8. Visual inspection at 90 days for (a) M1, (b) M2, (c) M3, and (d) M4

(a) (b) (c) (d)

Figure 9. Visual inspection at 180 days for (a) M1, (b) M2, (c) M3, and (d) M4

4.3.2. Total amount of chloride by binder weight

Preconditioned concrete samples were immersed in a chloride-rich environment to assess their resistance to chloride penetration based on 28, 90, and 180 days of exposure. Powdered samples were drilled at depths of 2 cm, 4 cm, 6 cm, and 8 cm and analyzed for the total chloride content using the Volhard titration technique. Figure 10 shows the chloride content as a percentage of the weight of the binder for samples M1, M2, M3, and M4 for various exposure times. At 28 days, the chloride content in the surface vicinity (2 cm) was 0.16%–0.23%, indicating greater chloride penetration in M2 than in the other mixes. The content of chloride decreased with depth gradually, and at 8 cm it was 0.07%–0.11% in all mixtures. This trend suggests that the exterior surface is most susceptible to chloride attack, whereas the interior layers are more protected because of microstructure densification. At 90 days, the amount of chloride at a 2 cm depth varied between 0.1% and 0.3%, whereas at 8 cm it was stable at 0.1%, indicating that the deeper layers were relatively immune to long-term exposure. At 180 days, chloride concentration at 2 cm was between 0.18% and 0.32%, affirming the steady accumulation of chloride over time. In every instance, the chloride content decreased with depth, highlighting the diffusion-controlled ingredients.

Among all the mixes, M3 (2% nano-silica + 20% micro-silica) exhibited the lowest chloride content at all depths and exposure times. The combination of nano-silica and micro-silica enhanced the matrix densification and refined the pore structure, significantly reducing chloride permeability. The amorphous nature and high surface area of nano-silica facilitated additional C–S–H gel formation, which effectively blocked chloride transport paths. On the other hand, M4 (3% nano-silica + 30% micro-silica), despite having better microstructural fineness, had marginally more chloride content at 2 cm depth after 180 days is 0.32%, likely due to microcrack formation related to excess silica content and decreased workability.

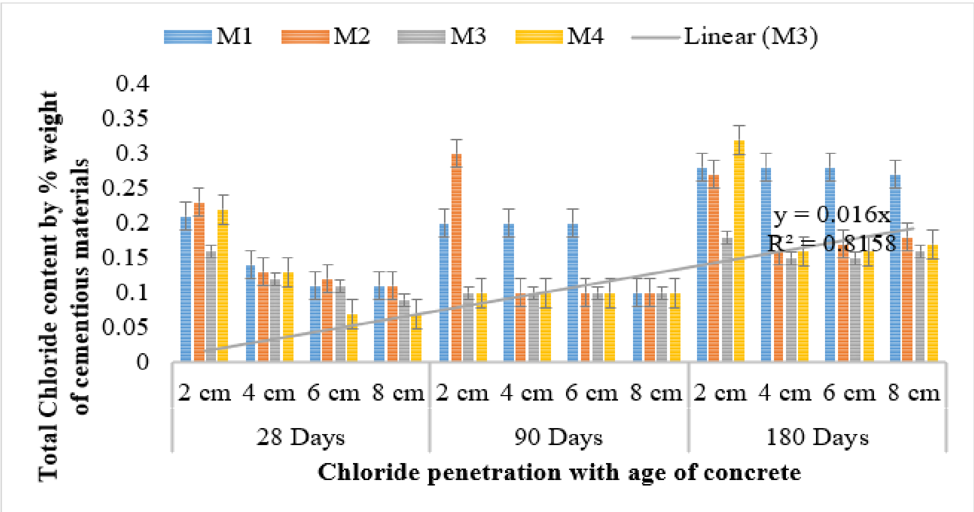


Figure 10. Total Chloride content by % weight of cementitious materials

The overall average chloride content for all mixes was less than the EN 206:2016 limit of 0.4% by weight of the binder, with exception being for M4 at 2 cm depth after 180 days. The general tendency of chloride penetration followed the sequence M4, M1, M2 and M3, which signified that M3 provided the best long-term resistance to chloride penetration. Chloride binding capacity depends on the occurrence of aluminate phases, which create Friedel's salt, trapping free chloride ions. Compositions containing nano and micro silica showed enhanced chloride binding attributable to the pozzolanic reaction and more detailed microstructure, raising the number of available binding sites.

Combined application of micro and nano silica enhanced the durability of the concrete by minimizing free chloride ions and facilitating stable chloride binding. Though M4 had the largest amount of micro silica, its over-replacement probably caused increased porosity and lower compaction, accounting for its higher chloride values at extended exposure times. Thus, M3 was best balanced in terms of strength as well as durability, validating that 2% nano silica and 20% micro silica offer better resistance to chloride migration, making it a sustainable, durable option for HPC structures.

5. STATISTICAL RELIABILITY OF EXPERIMENTAL RESULTS

All reported experimental values represent the average of multiple specimens tested under identical laboratory conditions in accordance with standard testing procedures to ensure repeatability and consistency of results. The observed performance trends among mixes M1, M2, M3, and M4 were consistent across all mechanical and durability tests, confirming the reliability of the experimental findings. Although formal statistical analyses such as ANOVA were not performed in the present study, the clear and systematic variations obtained across the tested parameters provide sufficient experimental evidence of the effectiveness of nano-silica and micro-silica incorporation. Future studies will include advanced statistical validation with larger datasets to further quantify the significance of these observed differences.

6. CONCLUSION

The laboratory experiment conducted to determine the impact of nano-silica and micro-silica on the mechanical properties and chloride penetration resistance of high-performance concrete reveals that substitution of cement with an optimal quantity of nano-silica and micro-silica has positive effects on mechanical strength and durability of concrete.

Tests with fresh concrete showed a steady decline in workability due to the increased water requirement, large replacement quantity caused by reduced densities of both materials relative to cement, and high specific surface area that lead to higher friction inside the mixture. However, all modified compositions possessed adequate workability, especially mixes M2 and M3. The lower values of slump and compaction factor together with differences in Vee-Bee time suggest greater cohesiveness of particles in the concrete mix.

In terms of mechanical performance, all modified mixes (M2–M4) exhibited higher compressive, split tensile, and flexural strengths than the control mix (M1). This improvement is attributed to the enhanced pozzolanic reactivity and filler effect of silica particles, as inferred from the observed experimental trends and supported by previous studies. The optimum performance was achieved by M3 (2% nano-silica and 20% micro-silica), which recorded a maximum compressive strength increase of 20.6% at 90 days compared to conventional concrete. Beyond this optimum dosage, as observed in M4, excessive silica content likely reduced effective cement hydration and increased water demand, resulting in strength reduction.

Chloride resistance results confirmed the beneficial influence of silica incorporation. $AgNO_3$ visual observations indicated lower chloride penetration depths in modified concretes. The mixes also exhibited improved chloride resistance, contributing to enhanced durability under aggressive exposure conditions. Volhard titration results showed chloride contents within the acceptable limits of EN 206:2016, except for M4 at 180 days (2 cm depth). Once again, M3 demonstrated the optimum balance between mechanical performance and chloride resistance.

The above discussion brings out the significance that lies in the use of both MS and NS in order to improve the performance characteristics of high-performance concrete. In this case, the best amount of both silica is at 2% NS and 20% micro-silica. The uniqueness of the present work is that there exists an experimentally proven best amount of the mixture that could be used in such an environment.

The current study confines itself to macroscopic assessment of performance only and has not undertaken any microscopic analysis of samples. Future research may consider employing SEM,X-ray diffraction, or FTIR spectroscopy to confirm the physics-chemistry behind the results. In addition, future research must investigate the effect of nano silica and micro silica along with other SCM's such as fly ash, slag, and metakaolin.

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IZVOD

ODRŽIVA PROIZVODNJA BETONA KORIŠĆENJEM RECIKLIRANIH AGREGATA I ADITIVA NA BAZI NANOTEHNOLOGIJA

U ovom radu ispituje se uticaj nano-silike (NS) imikro-silike (MS) na mehanička svojstva i otpornost HPC-a na hloride. Novina u ovomistraživanju je analizasinergizmaizmeđu NS i MS pucolane i pronalaženje optimalnog odnosa hibridnih doza. Da bi se sproveo eksperiment, proizvedene su četiri betonske mešavine, uključujući kontrolnu (M1) i tri modifikovane (M2, M3 i M4) zamenom veziva cementom. U M2, M3 i M4 procenti MS i NS u mešavinisu 1+10%, 2+20% i 3+30%, respektivno. U svežem stanju, betonske mešavine su okarakterisane testom sleganja, testom zbijanja i Vi-Bi testom. Za karakterizaciju u očvrslom stanju, merene su čvrstoće na pritisak, čvrstoće na zatezanje cepanje i čvrstoće na savijanje u starosti od 7, 14, 28, 56 i 90 dana. Za ispitivanje otpornosti na hloride, sprovedeni su $AgNO_3$ i Folhardovititracioni testovi nakon 28, 90 i 180 dana. Primećeno je da povećanje procenta silicijumdioksida dovodi do manje obradivosti svežeg betona, ali povećava čvrstoću i otpornost betona na hloride. Optimalni rezultat je dobijen sa M3, koji je zabeležio povećanje čvrstoće na pritisak od 20,6%, zajedno sa boljom otpornošću na prodiranje hlorida. Ovi rezultati pokazuju da je primena i NS i MS efikasna u poboljšanju strukturnih svojstava HPC-a.

Ključne reči: *Nano-silicijumdioksid, Mikro-silicijumdioksid, Visokoperformansni beton, Otpornost na hloride, Pucolanska reakcija*

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